

CHARGE NUMBER: 2525
PROJECT TITLE: Chemistry & Isolation of Tobacco
Constituents
PERIOD COVERED: July 1 - 31, 1980
PROJECT LEADER: S. A. Haut
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A systematic calibration of the Berthold HPLC radiochemical detector is now underway. Although this detector can be used with any form of HPLC it has been connected to our HPGPC so that the columns may be calibrated at the same time. The flow rate in preliminary data is the most influential factor in the radiochemical sensitivity of this instrument. ^{14}C -toluene and ^{14}C -dotriacontane have been used so far as reference compounds.

Our participation with Project 2501 in the Polonium study continues.²

The WS-14 synthesis work has begun to show some definite trends. The preparation of the Grignard Reagent (2-menthyl magnesium chloride) is now quite dependable and can be formed in essentially quantitative yield in refluxing THF. Reaction of t-butyl isocyanate with this reagent at low temperature (-78°C) tends to give a higher ratio of WS-14 to neo-WS-14 (ca. 9:1). Unfortunately the reaction is not instantaneous as it is at -5°C and if quenched after several hours overall conversion to products is only 30%. If the mixture is allowed to warm to room temperature the product ratios are identical to that at -5°C . Addition in refluxing THF increases the yield (81%) and the relative amount of neo isomer (2:1)³.

The studies directed towards developing preparative HPLC isolations of key components in complex natural products continues. Although we had in the past easily isolated such materials menthol and neomenthol from peppermint oils, the isolation of various other components from browning mixtures or tobacco extracts have proven more difficult. These latter materials will undoubtedly still require preliminary simplification.⁴

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The application of HPLC to work of the Chemical Research Division has begun. Already several members of the division outside our project are skilled in the use of the analytical HPLC's.

Some in depth separation development has been accomplished this month with respect to the purification of several grams of compounds 7415-89-1⁵ and 7462-19 for Project 2500. Preparative separations were performed on the following samples: 7415-89-1 (9 g), 7386-36a (4 g), 7446-46 (40 g), and 7462-7 (4.5 g).⁶

REFERENCES:

1. Chavis, M.
2. Barlow, K.
3. Haut, S.
4. Edmonds, M.
5. Brady, J.
6. Core, M.



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